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By

Dain A. Nelson

A Project

Submitted to the

Department of Packaging Science

College of Applied Science and technology

In partial fulfillment of the requirements for the degree of

Master of Science

Rochester Institute of technology

August 2nd, 2011

Department of Packaging Science
College of Applied Science and technology
Rochester Institute of Technology
Rochester, New York

CERTIFICATE OF APPROVAL by
Deanna Jacobs - Graduate Program Chair School of Packaging Science
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M.S. DEGREE PROJECT

**Implement modified version of Nestle oil test on flexible packaging
film to evaluate migration of Essential Oils on films coated with
layered nanoclay platelets.**

The M.S. degree project of Dain A. Nelson
has been examined and approved
by the above project committee as satisfactory
for the requirements for the
Masters of Science Degree

Abstract

Project followed a previous research method established by Nestle Purina for test of grease migration in Pet Food packaging using chicken fat and oleic acid. This procedure was modified and replaced with Essential oils commonly found in spices and seasoning blends. The objective was to observe the barrier effectiveness of an emerging coating technology (Nanoseal) using less than one micron thick layer of exfoliated clay platelets coated on thin packaging film commonly used in food packaging. Use the Nestle method to track Migration of Essential oils detectable under natural and UV light exposed to chromatography plates under ambient and elevated temperature with direct pressure applied to the test film surface. High temperatures in the procedure lead to evaporation of the oil in early test phases. NanoGold trace element later added to the oil as another tool to help track permeation in the film and clay platelet layer. The gold trace element as a marker proved inconclusive.

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Introduction

Contributors to this study are Frontier Natural Co-Operative a natural, organic whole sale foods company and NanoPack, Inc. NanoPack provided their NanoSeal TM vermiculite clay platelet barrier coated on films. Frontier provided aggressive plant based Essential Oils. Frontiers objective: obtain an effective film lamination structure for use with a wide range of seasonings, spice and aroma therapy products packaged in small pouches at the same time look at sustainability and recycle content of the total package. Previous pouch structures used had been either metalized polyester or paper film foil based structures. Ultimately Frontier wants to achieve a package structure with performance properties close to metalized Polyester or foil but also strive for a softer environmental footprint.

The use of exfoliated clay platelets of Nano thickness in one dimension deposited as a coating with a total layered thickness of less than one micron is believed will impart significant barrier to oil migration due to the presence of both layered platelets and the PVOH aqueous chemistry used to carry the platelets. Heat used to drive off the carrier resin enhances the coating durability through cross linking. The proprietary chemistry that carries the platelets keeps the platelets from agglomerating by neutralizing the positive charge on the platelets. The chemistry reestablishes negative charge on the platelet when a platelet is broken or sheared. A platelet sheared in processing exposes a positive charge at the brake point on the platelet. The carrier chemistry creates a regular Vander Waals gap between the platelets which enable the platelets to stay singular. The coating in this study is referred to in name as NanoSeal TM.

Background

In nanotechnology, a particle is defined as a small object that behaves as a whole unit in terms of its transport and properties. It is further classified according to size: In terms of diameter, fine particles cover a range between 100 and 2500 nanometers, while ultra fine particles, on the other hand, are sized between 1 and 100 nanometers. Similarly to ultra fine particles, **nanoparticles** are sized between 1 and 100 nanometers, though the size limitation can be restricted to two dimensions. Nanoparticles may or may not exhibit size-related intensive properties that differ significantly from those observed in a fine particles or bulk material. [Wikopedia]

The European Commission 2011 published its recommendation for defining a nanomaterial as follows...” A natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50% or more of the particles in the number size distribution, one or more external dimensions is in this size range 1nm – 100 nm. In specific cases and where warranted by concerns of the environment, health, safety or competitiveness the number size distribution threshold of 50% may be replaced by a threshold of 1 to 50%. By derogation from the above, fullerenes, graphene flakes, and single wall carbon nanotubes with one or more external dimensions below 1nm should be considered as nanomaterial.”

The Vermiculite Association, Lincoln UK states Vermiculite is a member of the phyllosilicate mineral group and is micaceous in nature. [Vermiculite Association] Mined from deposits formed 1-1/2 to 3 Billion years ago in many parts of the world; United

States, South Africa, China, Brazil and Zimbabwe. A limited number of sites are worked commercially. [Smith]

Vermiculite is most commonly used in its exfoliated form. The exfoliated vermiculite has a low bulk density, (light weight), absorbent, non-combustible and proven useful in many environmentally beneficial and public safety applications such as. [Vermiculite Association]

- Commercial and residential fire protection
- Growing media and soil amendments for fruits, vegetables and flowers
- Lightweight concrete to improve insulation and noise absorption
- Animal feeds to preserve integrity of feed
- Bio remediation aid to clean contaminated soils
- Packaging aid. Dispersion in composite polymer of barrier coating

Global markets for Nanotechnology will grow impressively in the next five years stated Pat Reynolds Editor for Packworld.com (March, 2010) and are expected to reach \$4,117,000.00 by 2015. Applications for Nanotechnology include Personal care, Cosmetics, Household care, Packaging and Leisure Wear and Equipment. [Reynolds]

In terms of the amount of nanomaterials consumed, papermaker's use of nanoparticles (NOs) is arguably the world's most important present application of nanotechnology. Worldwide, papermakers employ colloidal silica and related products each year to promote dewatering and fine-particle retention on hundreds of paper machines during production of over ten million annual tons of paper and paperboard products. [Hubble]

Exfoliated vermiculite clay referred to in this study is clay platelets 1-3 nanometers thick and 10-30 microns in breath, yielding an average aspect ratio of ~10,000:1. The platelets are maintained in a singularized format when dispersed into a PVOH resin and aligned in the plane of the coating when deposited on a film substrate, such as polyethylene terephthalate (PET). The dispersed platelets create a tortuous path of 'nano-bricks' not one but many layers that inhibit the movement of gas molecules. A water based resin coating system containing polyvinyl alcohol (PVOH) and other chemistries with wide aspect ratio platelets of vermiculite form the layered torturous path. Typical coating thickness is 1/3 of a micron or 300 nanometers. The PVOH resin and associated chemistry fills in the gaps between platelets. (Kravtiz)

Nano-Composite vs Nano-Coating

While nanotechnology can be lumped together for general purposes, there are four distinct divisions: nanoclay, carbon nanoparticles/ nanotubes; nanoscale metals and oxides; and biobased nanocomposites. The largest application in packaging comes from nanoclay or polymer-clay nanocomposites. (Fisher)

The ex-foliated clay commonly used in nanocomposites is organically modified montmorillonite. The concept of nanocomposites is to achieve much with little. With such benefits as heat resistance, barrier, strength, stiffness and flame resistance. [Pardos]

The key component of nanocomposites is silicate nano-clay with many mica-like platelets of 1nm thickness. The platelets are chemically treated to make them organophilic, meaning that polymer will enter the space between the platelets. The results are a nanocomposite polymer with many improved properties. [Pardos]

Image [2] shows polypropylene film with embedded nanoparticles of clay (dark objects). The inclusion of these nano-platelets significantly reduces oxygen diffusion in the film. The image shows ~ 100 nm sheets of clay.

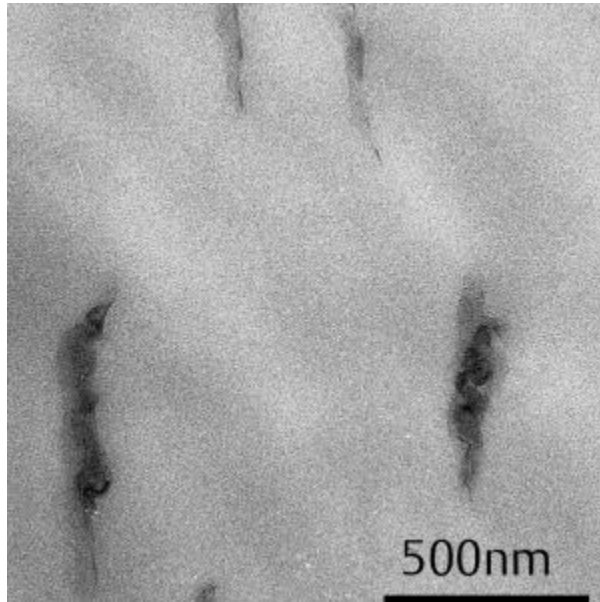
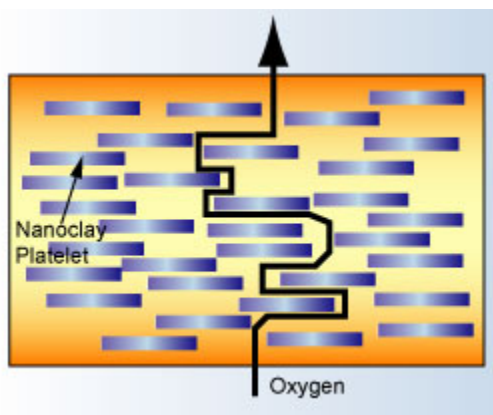


Image 2

Sample image prepared by Marilena Pezzuto, Institute of Chemistry and technology of Polymers in pozzuoli, Italy.

The use of nanocomposites has some draw backs. Wide gaps are possible between individual clay platelets with dispersion of the platelets in irregular alignment in the polymer.

Texas A&M University Department of Mechanical Engineering report on Layer-by Layer assembly of thin oxygen barrier using clay platelets. In this study the aspect ratio of the clay platelets were over 100 nm in diameter and 1 nm thick. Premise of this study to create a torturous path for gas molecules moving through the polymer matrix. In the study a 30 – bi-layer coating was created, 570 nm thick this very controlled method of platelet application resulted in OTR barrier <0.005 cc/m²/day atm. [Jang...]



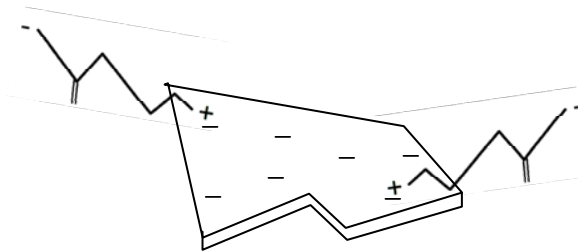
A tortuous path through the layered coating prevents gas molecules from passing in or out. Early commercial application of a nanoplatelet *coating* technology was with a company called InMat with their Air D-Fense 2000 and 3000 coating with tennis balls. Later NanoPack, Inc. developed their trademark two part NanoSeal barrier coating designed with a proprietary chemistry allowing platelets to stay singular. More importantly, commercial applications became viable with converters when the NanoSeal TM coating was applied and dried using on site coating and printing assets at economical run speeds.

NanoSeal Coating Attributes

NanoSeal is effective against Oxygen and Aroma transfer. Numerous and repeated testing of OTR, oxygen transition rates had been repeated with consistent data showing a dense layer of clay platelets applied to thin films produced impressive OTR barrier data. For example 70gauge OPP with 1/3 micron coating of NanoSeal TM was 1.0 – 1.6 cc/100 in²/ day. 48G PET with 1/3 micron coating of NanoSeal TM was at 0.10 – 0.30 cc/100 in² / day. (Kravitz)

NanoSeal can also impart additional benefits to food packaging in the area of tarnishing of flavors. Food packaging, although an integral part of the food chain, has a major drawback in that, often, the packaging material interacts with the flavor constituents of the food, causing either a selective or an extensive loss of desirable food flavors or absorption of undesirable off-flavors from the packaging material, thereby resulting in an eventual loss of quality of the packaged food item. The process is called “scalping” [Sajilata] Reduction in this gas or aroma transfer inside and outside the package will aid in product preservation.

Aroma Barrier: Product Technology



Aroma barrier is a new concept and use for NanoSeal coatings. The negatively charged clay particles in the NanoSeal coating attract the positively charged ends of polar molecules of aroma and flavor molecules.

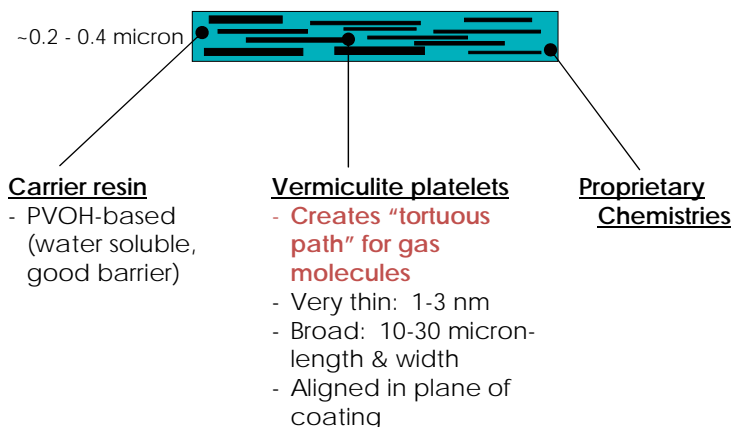
NanoSeal TM is available in a two part system; aqueous dispersion of vermiculite platelets at approximately 8% solids and ‘master batch’ of polyvinyl alcohol (PVOH) resin with proprietary chemistry that keeps the platelets from agglomerating and

improves moisture –resistance of the PVOH resin. The two parts are mixed gently under ambient conditions just prior to applying the coating. [Kravitz]

The key technical difference between NanoSeal coatings and past attempts to use nanoclays for barrier enhancement is that with NanoSeal the clay platelets remain in singularized format not only when dispersed in the resin, but also during and after deposition. “Barrier properties require the greatest degree of dispersion and exfoliation, in which the clay platelets must be both separated and aligned to create a tortuous path that is a barrier to gases and chemicals.” (Markarian, Fisher) Not only does this achievement enable for the tortuous path required for high gas barrier, but it also enables cohesive strength necessary for multilayered packaging film structures. [Kravitz]

PVOH a carrier resin for the platelets is considered environmentally friendly resin and has widespread use as the adhesive of choice in corrugate box board. This resin with vermiculite is applied to a coating thickness of 0.2 to 0.5 microns.

NanoPack’s Barrier Coating



This study intended to add a new body of knowledge to known gas and moisture barrier already established with NanoSeal data points collected. The NanoSeal coating on various films will be subjected to temperature and pressure to test the effectiveness as a barrier to essential oils such as those found in spices and aroma therapy products.

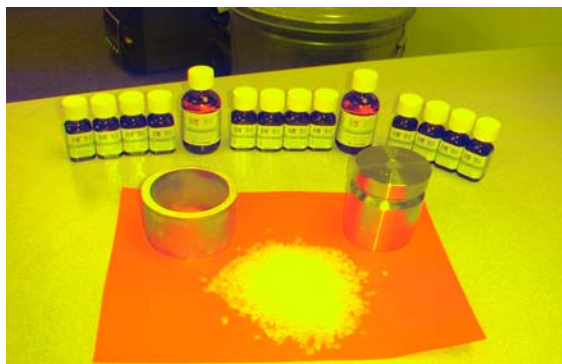
Project Methodology:

Modified version of study published in 2004 by the Nestle Purina Research Center packaging laboratory in Switzerland titled a “Novel Method for Testing the oil and Grease Resistance of Plastic Based Dry Pet Food Packaging”. The Nestle study developed the use of Chromatography 10 cm x 10 cm plates placed under a film sample which made it possible to visualize fat or grease migration under UV light. Study determined optimum test conditions at 70C with dry air, test duration 16 hours. The Chromatography plates exhibit fluorescence, emit green light when illuminated with UV light at 245nm, this light is suppressed when UV absorbing liquids such as the fats come in contact with the Chromatography plate. RIT center for Imaging Science will offer help to show if the essential oil is blocked by the surrounding PVOH resin, or evidence of oil migration around the platelets.

The research group at Nestle saw the need for a test method for plastic packaging that was close to the method established for paper packaging. The conclusion at Nestle was to establish a new test condition, 70 C and dry air. Test duration 16 hours. Nestle research found the test procedure gave correct results for all types of plastics and minimal sample to sample variation.

With this test method established by Nestle Purina for Grease resistance for plastic based dry pet food packaging the Nestle procedure was modified. Essential oil will replace chicken fat and oleic acid. The Essential oils supplied by Frontier Natural Co-operative are: Vetiver, Clove Bud, Eucalyptus, Peppermint and Grapefruit.

Essential Oils and Materials



Polyester films coated and uncoated with NanoSeal

Round ½” thick Glass plate to carry and rest test material in the bench oven.

Modified rubber gasket found at local hardware store. Prevent oil migration from under

Stainless Steel Ring (made from Wall Tube 304 stainless) 2.5” ID x 3.0 OD x 25” x 2.5”

Thin HPTLC Chromatography glass plate dimension 10 cm x 10 cm

Stainless Steel weight Type 303 Easy Grip Test weight 2KG / 2,000 grams

Bench Oven from Quincy labs Model 21-250

Sigma- Aldrich Silica gel 3-9 Mesh 214434 – 1 KG

Triton T2 Digital Pocket scale

2 ml capacity dropper

Chromatography Viewer. Camag UV cabinet II. Dual wavelength 254/366 nm.

Acquired from Lab Equip Ltd, Markham, ON Canada

NanoGold Crystals concentration of 10 mg nanogold element in 5 ml none polar solvent solution. Supplied by N& N labs at the University of Arkansas

Test Reiterations.

Examples of staining on Chromatography plates:

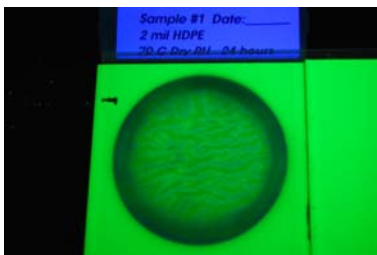
Phase 1 testing with Citrus and Clove bud oils at 70C – 16 hrs, 24 hours and 48 hours

Phase 2 testing with Citrus oil with NanoGold Crystals

Phase 3 ALL essential oils tested. Bench oven temperature reduced to 50C pulled vacuum on samples to prevent evaporation.

Phase 4 test Vacuum sealed ran test at ambient room temperature

The Nestle research used graph paper and chromatography viewer to observe migration of oils and staining. The Nestle procedure was followed and coated film samples were selected and exposed to temperature and pressure to observe results of oil migration to obtain a visual of the result.



Phase 1 test



70C- 16 hours: Frontier Clove oil and Citrus oil selected, oven temperature set at 70 C. Temperature condition one Kilogram weight and 60 mm stainless steel ring. Place white sheet of graph paper on heavy glass plate. 48G Polyester sample film selected and cut to 14 cm x 11cm size and placed over the white graph paper. Weigh 40 grams of silica gel in 50 ml capacity glass beaker, pour silica into stainless steel ring which was placed on a heavy glass plate. 4 ml of Clove oil mixed in the silica gel, a 60 mm diameter stainless steel ring positioned at the center of the sample PET film, mixture of Silica gel and clove oil placed inside the ring. One kilo weight placed inside the ring resting on the silica gel. Made three (3) tests, Sample A, B and C of the identical test set up placed in the bench oven. Observed test A material after 16 hours no stain on the graph paper. No visual sign of oil migration or stain. Observed test B after 26 hours same result no Stain detected. Removed test C after 48 hours and again no sign of stain on the graph paper.

Phase two testing

Used Grapefruit Oil (Citrus). Same procedure followed in first test.

Test sample A - 48G PET plain No coating. Three sets of samples prepared. Test sample B - 48 G PET with PVOH no Platelets. Test sample C - 48 G PET PVOH with Platelets. Graph paper checked for each test A, B and C. After 16 hours, 24 hours and 48 hours. No stain observed. Conclusion, per discussion with Dr, Ravin Donald the high temperature of the bench oven at 70C was affecting the test procedure by dry off the essential oil before the oil had an time to migrate. Suggest placing the test in a vacuum with addition of trace element for tracking the coating migration.

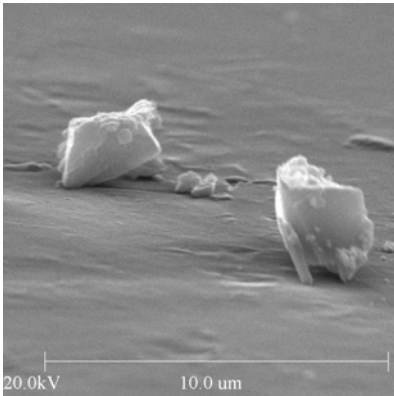
Repeated the test with Citrus (PET with coating no platelets) and Clove oil (PET film only) and extended the time to seven days in the bench oven. Observations made showed no signs or migration or stains on the graph paper under the PET. The Clove oil in the silica gel was cranberry in color, dry to the touch. The Citrus oil showed no signs of stains on the graph paper. Appearance of the Silica gel was yellow in color like lemon.

I reported my observation to Dr. Ravin Donald at Frontier. His reply back was the volatile portions would definitely evaporate at 70 C in fact even at room temperature this will be the result. Room temperature testing would be best. Addition of more oil would not help. A closed system to test like vacuum sealing would help keep the volatile compounds close to the film and coating for a longer time.

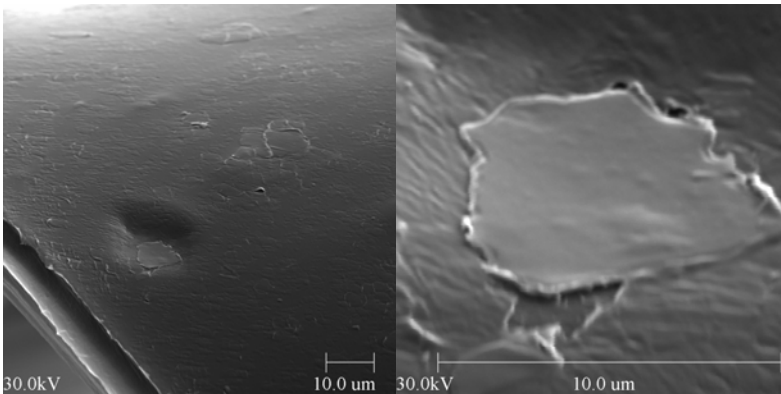
First visit to RIT Imaging Lab

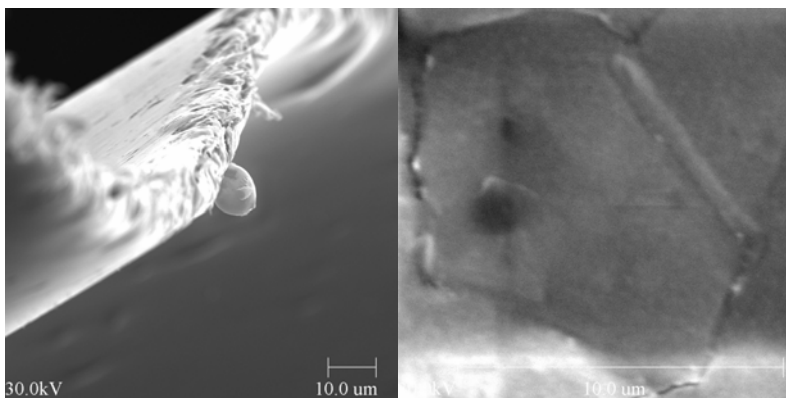
Two film samples of PET with nanoseal were prepared with platinum –silver coat for imaging with the SEM. First viewing at the Nano-Imaging lab of nanoseal coating on PET we were not sure what we were viewing a platelet, surface contamination or both.

This image shows silica gel contamination on the surface of the PET film.



With increased power using the SEM scanning electron microscope for more magnification the beam would begin to degrade the sample. The images obtained show vermiculite platelets on the surface of the PET film, platelets with both rough and smooth edges observed.

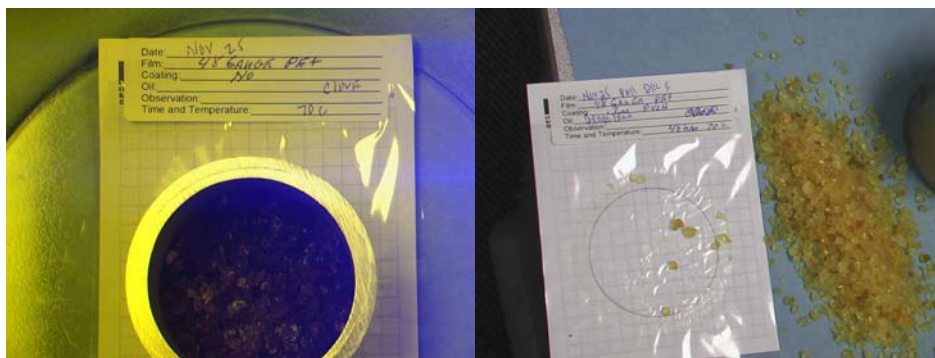




Suggestion made by Dr. Hailstone to help with identification. Create profile image of dry platelet to compare with SEM images

Phase 3 test Procedure and with follow up at RIT Nano Imaging lab

This test phase used Citrus oil (Grapefruit) Oil and Clove bud oil and prepared as in procedure outlined in Phase one with addition of 0.25ml and 0.50ml of *nanogold crystals* to 4 ml citrus oil as a trace element maker to help identify extent of oil movement or migration through the film and nanoseal coating. Samples with Nanogold element planned for SEM imaging at RIT. Clove oil test was with PET film. Observed the silica gel very dark (cranberry in color). Silica was dry with no sign of oil residue. Both the Clove and citrus oils has dissipated over the time period. These samples prepared with the nanogold element taken to RIT for imaging.



Three (3) samples prepared. A) PET film uncoated 2) PET Coated with PVOH and C) PET with PVOH containing Clay Platelets. Placed samples in vacuum sealed pouch per recommendation by Dr. Ravin Donald at Frontier used Rival vacuum sealer. Test not placed in the bench oven to avoid evaporation but left at ambient conditions for seven days, After seven days the test material evaluated. The silica gel had not discolored significantly and was dry, no oil residue. The aroma of Citrus slight and had mostly dissipated



Observation: The silica gel with Citrus oil very dark (cranberry in color). Silica was dry with no sign of oil residue. Both the Clove and citrus oils has dissipated over the time period. Close examination of the graph paper under the PET film sample showed signs of some staining. The stain is a faint red in color samples prepared with the nanogold element taken to RIT for imaging.

TEM Image [1] taken at Rochester Institute of Technology, Rochester, NY courtesy Dr. Rich Hailstone., Co-Director Nano-imaging Lab Images show vermiculite overlapping platelets 68,000 magnification. Samples prepped from a 10,000 x dilution of a solution with distilled water. Solution provided by NanoPack, Inc.

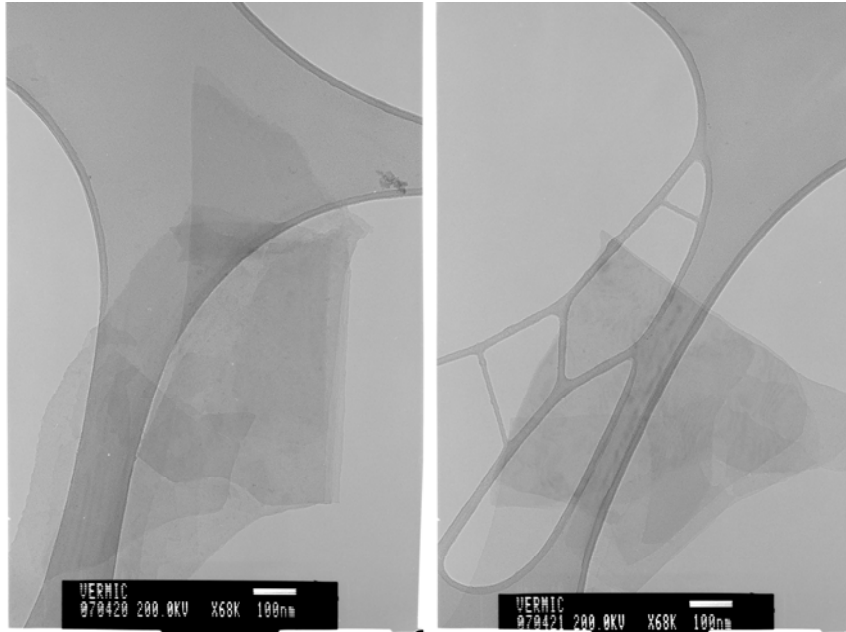


Image 1

Solution with exfoliated vermiculite deposited on a 3mm diameter “grid” which is just a very fine mesh copper screen on which a “holey” carbon film deposited. The curved structures in the image show where the carbon film is at. The platelets partly extend over the open area of the grid, where the electron beam will only go thru the platelet and the image in that area is not compromised by the carbon background. Both images show overlapping platelets of irregular dimensions. Suggestion by Dr. Hailstone, Element profile of vermiculite and Element marker [] with a different profile than the organic oils would be helpful. Dr, Ravin Donald from Frontier had experience with

Sulfur and Gold elements with SEM and recommended nanogold for use with the essential oil matrix as it would be easier to distribute in the essential oil.

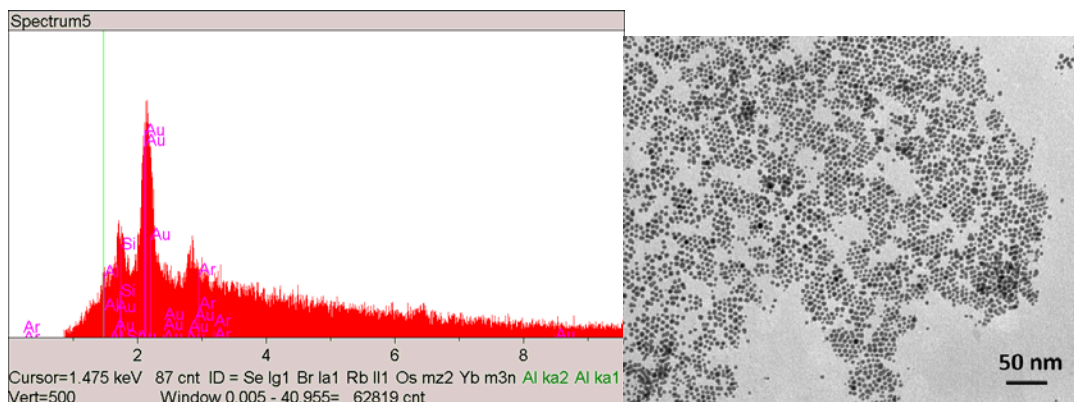


Image on the left Element Spectrum analysis surface scan of Nanocoated PET film with Nanogold add to Essential Oil. Image to the right is of the nanogold element added to the essential oil supplied by N&N Labs, University of Arkansas

Follow up to this project study.

Supplemental analysis done by Dr. Ravin Donald from Frontier Natural Co-operative gave additional confidence in the NanoSeal layered Clay platelet barrier packaging option by conducting a steam volatile oil (SOV) test using several essential oils.. The film structure used 48 gauge PET / Nanoseal coating / adh / 2.0 mil cast polypropylene sealant, CPP. Test conditions: Ambient + 20 degrees F. No humidity control. Organoleptic results reported after eleven weeks.

Slight flavor profile reduction, however, flavor reported as typical. Film structure stability after eleven weeks still held with very slight amount of de-lamination observed with Ground Cloves.

Steam Volatile Oil is a measure of volatile oil percentage in herbs and spices (given as a v/w %). Example, A 100g sample of cloves with SVO level of 18% would have a 18 ml of volatile oil.

Steam Volatile oil is determined by a simple heating of the ground product in H₂O and using a condenser to collect the volatilized fraction. Some variance in testing can be +/- 5%, so the values seen over time were essentially stable. The conclusion from Dr. Donald SVO evaluation with NanoSeal in the film structure was essentially no change in the flavor component. Steam Volatile Oil results (%) in Frontier's evaluation are the following. Courtesy: NanoPack Inc. and Frontier Natural Co-op.

Aroma Barrier: Steam Volatile Oil Results

Sample/Time	% Oil Retained		
	0	3 weeks	11 weeks
Cloves, whole	100.0%	95.6%	95.6%
Cloves, ground	100.0%	100.0%	100.0%
Cinnamon	100.0%	100.0%	100.0%
Peppermint	100.0%	89.7%	79.3%

ASTA 5.0

Evidence of aroma barrier is shown as the percent of flavor and flavor components lost during an 11-week, elevated temperature (120F) storage study. Common loss of aroma and flavor components under these storage conditions ranges from 25 to 50 percent.

Conclusion:

Use of modified Nestle method was effective, substituting Essential oils did not show migration in the form of stains on chromatography plates under UV light. High temperature at the start of the test caused evaporation; subsequent lower temperature still did not show detectable oil migration. At ambient temperature detection of oil migration was still not evident. Placement of test materials in a vacuum was attempted to mitigate evaporation, to pull a vacuum on the test material proved a challenge with disruption of the test material while pulling a vacuum. The question remains open as to a conclusive role platelets have in retarding migration of essential oils. The PVOH resin and other chemistries that surround the platelets appear to provide a holdout benefit to oil migration without conclusive proof.

Additional opportunities to use the test method can be explored using bio-based films with and without platelets in the coating. Another area to test is platelet load or ratio of platelets in suspension in the PVOH carrier resin.

Off-odor is another area for study, the effectiveness of the Nanoseal coating to block out sources of odors coming from fungi, bacteria and microbiological sources.

Photo courtesy NanoPack Inc.

NanoSeal barrier coating in retail packaging.



Photos supplied by Nelson Packaging Sales, Inc.

Nanoseal in wholesale and end use packaging.



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